

CLAIMS

1. A process for producing a poly(arylene sulfide) by polymerizing a sulfur source and a dihalo-aromatic compound in the presence of an alkali metal hydroxide in an organic amide solvent, which comprises:
- (1) a dehydration step of charging, into a reaction vessel, the organic amide solvent and the sulfur source including an alkali metal hydrosulfide, and a part of an overall charged amount of the alkali metal hydroxide as needed, and heating a mixture containing these components to discharge at least a part of a distillate containing water from the interior of the system containing the mixture to the exterior of the system, and
- (2) a polymerization step of mixing the mixture remaining within the system after the dehydration step with a dihalo-aromatic compound, heating a mixture for polymerization reaction containing these components to subject the sulfur source (hereinafter referred to as "available sulfur source") and the dihalo-aromatic compound to a polymerization reaction, and adding the alkali metal hydroxide to the mixture for polymerization reaction continuously or in portions to control the pH of the mixture for polymerization reaction within a range of from 7 to 12.5 from the beginning to the end of the polymerization reaction.

2. The production process according to claim 1,
wherein in the dehydration step, the alkali metal
hydrosulfide or a mixture of at least 51 mol% of the alkali
metal hydrosulfide and at most 49 mol% of an alkali metal
sulfide is charged as the sulfur source including the
alkali metal hydrosulfide.

3. The production process according to claim 1,
wherein in the dehydration step, a mixture of 97 to 99.5
mol% of the alkali metal hydrosulfide and 0.5 to 3 mol% of
an alkali metal sulfide is charged as the sulfur source
including the alkali metal hydrosulfide.

4. The production process according to claim 1,
wherein in the dehydration step, the mixture is heated to
100 to 250°C to discharge at least a part of the distillate
containing water from the interior of the system containing
the mixture to the exterior of the system.

5. The production process according to claim 1,
wherein in the polymerization step, the alkali metal
hydroxide is added to the mixture for polymerization
reaction continuously or in portion to control the pH of
the mixture for polymerization reaction within a range of
from 9 to 12.1 from the beginning to the end of the
polymerization reaction.

6. The production process according to claim 1,
wherein in the polymerization step, the alkali metal
hydroxide is added to the mixture for polymerization
reaction continuously or in portion so as to satisfy the
5 following expression (I):

$$0 \leq y - x < 1.1 \quad (I)$$

wherein $y = \Sigma \text{OH}/\text{available S}$ (ΣOH being a cumulative molar
amount of a molar amount of an alkali metal sulfide
contained in the sulfur source charged in the dehydration
10 step, a molar amount of the alkali metal hydroxide added in
the dehydration step, a molar amount of hydrogen sulfide
volatilized out of the system in the dehydration step and a
molar amount of the alkali metal hydroxide added
continuously or in portions in the polymerization step, and
15 the available S being a molar amount of an available sulfur
source contained in the mixture remaining in the system
after the dehydration step), and x is a consumption rate of
the dihalo-aromatic compound, i.e., (a molar amount of the
dihalo-aromatic compound consumed in the polymerization
20 step)/(a molar amount of the dihalo-aromatic compound
charged)].

7. The production process according to claim 6,
wherein the alkali metal hydroxide is added to the mixture
25 for polymerization reaction continuously or in portion in
such a manner that the $(y - x)$ value in the expression (I)
satisfies a range of from 0 to smaller than 0.2.

8. The production process according to claim 1, wherein in the production step, the polymerization reaction is conducted by an at least two-stage polymerization process comprising:

- 5 (A) Step 1 of heating the mixture for polymerization reaction to 170 to 270°C in the presence of water in a proportion of 0.0 to 2.0 mol per mol of the available sulfur source to conduct a polymerization reaction, thereby forming a prepolymer that a conversion of the dihalo-
- 10 aromatic compound is 50 to 98%, and
- (B) Step 2 of controlling the amount of water in the mixture for polymerization reaction so as to bring about a state that water exists in a proportion of 2.0 to 10 mol per mol of the available sulfur source, and heating the
- 15 reaction system to 245 to 290°C, thereby continuing the polymerization reaction.

9. The production process according to claim 1, wherein in the production step, the dihalo-aromatic

20 compound is mixed in such a manner that the amount of the dihalo-aromatic compound charged falls within a range of from 0.9 to 1.50 mol per mol of the available sulfur source.

10. The production process according to claim 9,

25 wherein in the production step, the dihalo-aromatic compound is mixed in such a manner that the amount of the dihalo-aromatic compound charged falls within a range of

from 1.00 to 1.09 mol per mol of the available sulfur source.

11. The production process according to claim 1,
5 wherein in the dehydration step and production step, the overall charged amount of the alkali metal hydroxide is controlled in such a manner that the Σ OH value (i.e., a cumulative molar amount of a molar amount of an alkali metal sulfide contained in the sulfur source charged in the
10 dehydration step, a molar amount of the alkali metal hydroxide added in the dehydration step, a molar amount of hydrogen sulfide volatilized out of the system in the dehydration step and a molar amount of the alkali metal hydroxide added continuously or in portions in the
15 polymerization step, and the available S being a molar amount of an available sulfur source contained in the mixture remaining in the system after the dehydration step) falls within a range of from 1.0 to 1.1 mol per mol of the available sulfur source.

20

12. The production process according to claim 11,
wherein in the dehydration step and production step, the overall charged amount of the alkali metal hydroxide is controlled in such a manner that the Σ OH value falls
25 within a range of from 1.02 to 1.08 mol per mol of the available sulfur source.

13. A poly(arylene sulfide) obtained by polymerization in an organic amide solvent and having a nitrogen content of at most 800 ppm.

5 14. The poly(arylene sulfide) according to claim 13, wherein the nitrogen content is at most 600 ppm.

15 15. The poly(arylene sulfide) according to claim 13, wherein the melt viscosity thereof is 10 to 1,500 Pa·s as measured at a temperature of 310°C and a shear rate of 1,216 sec⁻¹.

16. The poly(arylene sulfide) according to claim 13, wherein in a melt viscosity range of the poly(arylene sulfide) from 140 to 300 Pa·s as measured at a temperature of 310°C and a shear rate of 1,216 sec⁻¹ by controlling the resin pH to at most 6.0 before measuring the melt viscosity, the nitrogen content satisfies the following expression (II-1):

20
$$Y < -1.5X + 800 \quad (II-1)$$

wherein Y is a nitrogen content (ppm) in the resin, and X is a melt viscosity (Pa·s) of the poly(arylene sulfide), while in the case where the melt viscosity exceeds 300 Pa·s, the nitrogen content satisfies the following expression

25 (II-2):

$$Y < 350 \quad (II-2)$$

wherein Y is a nitrogen content (ppm) in the resin.

17. The poly(arylene sulfide) according to claim 13,
which is a poly(arylene sulfide) obtained by polymerizing a
sulfur source and a dihalo-aromatic compound in the
presence of an alkali metal hydroxide in the organic amide
5 solvent.

18. A poly(arylene sulfide) having a ratio MVL/MVH
of a melt viscosity MVL (as measured at a temperature of
310°C and a shear rate of 1,216 sec⁻¹) of the poly(arylene
10 sulfide), whose pH is adjusted to at most 6.0, to a melt
viscosity MVH (as measured at a temperature of 310°C and a
shear rate of 1,216 sec⁻¹) of the same poly(arylene
sulfide), whose pH is adjusted to at least 8.0, ranging
from 0.7 to 1.0.

15

19. A poly(arylene sulfide) obtained by
polymerization in an organic amide solvent and having a
nitrogen content of at most 800 ppm and a ratio MVL/MVH of
a melt viscosity MVL (as measured at a temperature of 310°C
20 and a shear rate of 1,216 sec⁻¹) of the poly(arylene
sulfide), whose pH is adjusted to at most 6.0, to a melt
viscosity MVH (as measured at a temperature of 310°C and a
shear rate of 1,216 sec⁻¹) of the same poly(arylene
sulfide), whose pH is adjusted to at least 8.0, ranging
25 from 0.7 to 1.0.

20. The poly(arylene sulfide) according to claim 19,

wherein in a melt viscosity range of the poly(arylene sulfide) from 140 to 300 Pa·s as measured at a temperature of 310°C and a shear rate of 1,216 sec⁻¹ by controlling the resin pH to at most 6.0 before measuring the melt viscosity,
5 the nitrogen content satisfies the following expression (II-1):

$$Y < -1.5X + 800 \quad (\text{II-1})$$

wherein Y is a nitrogen content (ppm) in the resin, and X is a melt viscosity (Pa·s) of the poly(arylene sulfide),
10 while in the case where the melt viscosity exceeds 300 Pa·s, the nitrogen content satisfies the following expression (II-2):

$$Y < 350 \quad (\text{II-2})$$

wherein Y is a nitrogen content (ppm) in the resin.